FURACA #03/97 (N/15)

AIM: Preparation of Furaca from New route.

eq. to 100 gm. 2ACA.

I Preparation of TFA

RAW MATERIALS

2-Furoyl chloride	: 46.0ml
DMW	: (700 + 30)ml
NaSH (25% excess)	: 75.0gm
EtOAc	: 500.0ml
1:1 HCl	: 95.0ml
DMW	: 350.0ml
NaHCO3	: 39.0gm
EtOAc	: 200.0ml
1:1 HCl	: 82.0ml

Procedure

1. ~~Charged~~ DMW, NaSH were charged at RT - Stir to get clear solution (flush ⅓ 30ml DMW)
2. Add Furoyl chloride in 40-45' at 20-25°C
3. Stirred for 5', Monitor the reaction
4. Charged EtOAc, then brought pH to 1.0-0.9 by 1:1 HCl at 20-25°C in 10-15'
5. Separate the layers, Give sample of all
6. To that all added DMW, Adjusted to 7.0-7.2 pH by NaHCO3 at 20-25°C
7. Separated the layers
8. Added EtOAc to the ~~org~~ aqueous phase again brought pH to 1.0-0.9 by 1:1 HCl at 20-25°C
9. Separated the layers Sampled for each layers.
10. Org. phase ready for next step.

ii) preparation of Furaca

Raw materials

7ACA	: 103.0gm.	
EtOAc	: 400.0ml.	
HOAc	: 60.0ml	
BF_3	: 144.0gm	7 gm. extra BF Hence input adjusted
TFA	: 960.0ml	
Hydrolysis: DMW	: 300.0ml	
Hydrolysis: SHS	: 2.0gm.	
20% NH_3 Sol^n	: 184.0ml	
DMW	: (100 + 300 + 100) ml	washing
EtOAc	: (100 + 300 + 100) ml	washing
	(Slurry-wash)	

Procedure

1. charge 7ACA into the System having BF_3 purged in EtOAc + HOAc mixture at 20°C
2. Add TFA solution as prepared before
3. Maintain the temp at 30°C for 2hrs.

7ACA	Furaca Imp	TFA
2%/	3.	82.57%.
1hr 35'		2.

4. After completion of reaction, transfer the mass into the DMW (precooled at 15°C) with SHS
5. Adjust the pH to 3.5 by 20% NH_3 at 20-25°C in 25-30'
6. Stir for 30' at 20-25°C
7. Filter & Wash with DMW & EtOAc

NOTE:

Instead of 137.0 gm of BF_3, purged 144.0 gm of BF_3. So that added 10% for FACA.